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(54) EPOXY RESIN COMPOSITION AND SEMICONDUCTOR APPARATUS

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a high thermal conductivity epoxy resin composition which is characterized in that the composition is compounded with >90 mass %, based on the whole filler, of spherical cristobalite as an inorganic filler in an epoxy resin composition comprising an epoxy resin, a curing agent and the inorganic filler as essential components.

SOLUTION: This epoxy resin composition containing spherical cristobalite has excellent fluidity, slight abrasion of molds and provides a cured material having high thermal conductivity by making the composition include a specific amount of spherical cristobalite as the inorganic filler. Consequently, the epoxy resin composition containing the spherical cristobalite is suitably useful as a semiconductor sealing material even for an ultra-advanced device.

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TI Thermal conductive epoxy resin compositions having excellent flowability without abrading molds and semiconductor devices
IN Yoshino, Masachika; Takei, Minoru; Shiohara, Toshio
PA Shin-Etsu Chemical Industry Co., Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
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LA Japanese
IC ICM C08L063-00
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CC 38-3 (Plastics Fabrication and Uses)
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CLASS

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
JP 2001172472	ICM	C08L063-00
	ICS	C08K007-18; H01L023-29; H01L023-31
	IPCI	C08L0063-00 [ICM,7]; C08K0007-18 [ICS,7]; H01L0023-29 [ICS,7]; H01L0023-31 [ICS,7] <--

AB The compns. comprise epoxy resins, curing agents, and inorg. fillers containing ≥90% spherical cristobalite. Thus, a composition containing spherical cristobalite 530, crystalline silica (Cristalite 3K) 50, o-cresol novolak epoxy resins (EOCN 1020 55) 10, biphenyl epoxy resin (YX 4000H) 45, indene oligomer (IP 100) 5, novolak phenolic resin (TD 2131) 33, and brominated epoxy resin (BREN S) 7 parts showed spiral flow 91 cm and thermal conductivity 2.41 W/mK.

ST thermal conductive epoxy resin spherical cristobalite; novolak epoxy resin semiconductor packaging; flowability epoxy resin blend fused silica

IT Phenolic resins, uses
RL: MOA (Modifier or additive use); RCT (Reactant); RACT (Reactant or reagent); USES (Uses)
(novolak, crosslinking agents; thermal conductive epoxy resin compns. having good flowability without abrading molds and semiconductor devices)

IT Epoxy resins, uses
RL: POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); USES (Uses)
(phenolic, novolak, cresol; thermal conductive epoxy resin compns. having good flowability without abrading molds and semiconductor devices)

IT Electronic packaging materials
Semiconductor device fabrication
(thermal conductive epoxy resin compns. having good flowability without abrading molds and semiconductor devices)

IT 345341-77-7P
RL: IMF (Industrial manufacture); POF (Polymer in formulation); PRP (Properties); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)
(thermal conductive epoxy resin compns. having good flowability without abrading molds and semiconductor devices)

IT 14464-46-1, Cristobalite
RL: MOA (Modifier or additive use); USES (Uses)
(thermal conductive epoxy resin compns. having good flowability without abrading molds and semiconductor devices)

* NOTICES *

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] The fluidity of this invention is good, and there is little metal mold wear, it gives the hardened material which has high temperature conductivity, and relates to the semiconductor device by which the closure was carried out with the epoxy resin constituent containing the spherical cristobalite which can be suitably used also as latest encapsulant for a thin package etc., and the hardened material of this constituent.
[0002]
[Description of the Prior Art] In the resin constituent as the encapsulant for semiconductor packages, especially encapsulant for semiconductor devices containing a diode transistor, since a high current flows to a semiconductor device, generation of heat of a semiconductor device is large, and in order to miss this heat, the epoxy resin constituent which made thermal conductivity high by high restoration of a crystalline silica with high thermal conductivity is used. However, when high-filled up with the crystalline silica, shaping of transfer becomes impossible, and there was a fault, like the fluidity of a constituent worsens too much and metal mold wear becomes intense, and there was a limitation also in high restoration of a crystalline silica.
[0003] The attempt which is going to solve the above-mentioned trouble by mixing an alumina, especially a spherical alumina is performed to such a problem for many years. However, the being [the spherical alumina which does not contain the impurity of ionicity / easy to manufacture], and cost-problem has arisen.
[0004] It was made in order to solve the above-mentioned trouble, and a fluidity is good, there is little metal mold wear, and this invention aims at offering the semiconductor device by which the closure was carried out with the high temperature conductivity epoxy resin constituent containing the spherical cristobalite which gives the hardened material which has high temperature conductivity, and the hardened material of this constituent.
[0005]
[The means for solving a technical problem and the gestalt of implementation of invention] this invention person had the good fluidity by blending a spherical cristobalite exceeding 90 mass % of the whole bulking agent in the epoxy resin constituent which uses an epoxy resin, a curing agent, and a minerals bulking agent as an indispensable component as a result of repeating examination wholeheartedly, in order to attain the above-mentioned purpose as a minerals bulking agent, and there was little metal mold wear, and he found out that the epoxy resin constituent containing the spherical cristobalite which gives the hardened material which has high temperature conductivity was obtained.
[0006] That is, spherical fused silica is used for the current closure epoxy resin constituent for semiconductor packages so much, and the grain size, supply, and its unit price are also stable. By using so much what cristobalite-ized this spherical fused silica, the fluidity was good and it found out that the epoxy resin constituent which has high temperature conductivity with little metal mold wear was possible.

[0007] in this case, the epoxy resin constituent with which the whole bulking agent blended the spherical cristobalite one to 90% of the weight as a minerals bulking agent is proposed -- **** (Japanese Patent Application No. No. 122805 [ten to]) -- When a spherical cristobalite is blended exceeding 90 mass % of the whole bulking agent, a fluidity carries out the knowledge of it being good, there being little metal mold wear, and the formation of high temperature conduction with which the closure of the high exoergic type semiconductor device by the high current can also be presented enough being possible, and it comes to make this invention.

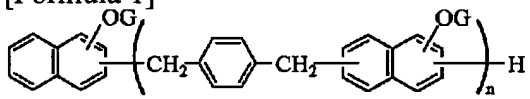
[0008] Therefore, the semiconductor device by which the closure was carried out with the high temperature conductivity epoxy resin constituent containing the spherical cristobalite characterized by this invention coming to blend a spherical cristobalite exceeding 90 mass % of the whole bulking agent in the epoxy resin constituent which uses an epoxy resin, a curing agent, and a minerals bulking agent as an indispensable component as a minerals bulking agent, and the hardened material of this constituent is offered.

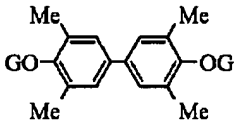
[0009] Hereafter, if this invention is explained to a detail, the epoxy resin constituent containing the spherical cristobalite of this invention will become considering an epoxy resin, a curing agent, and a minerals bulking agent as an indispensable component.

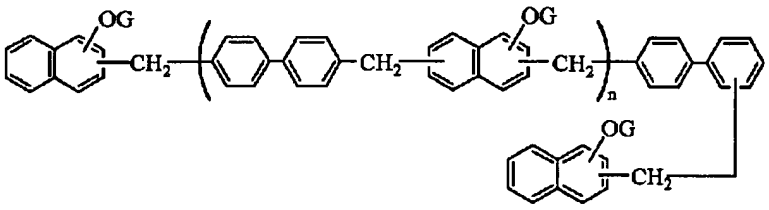
[0010] Anythings can be used if it has two or more epoxy groups per well-known monad from the former as an epoxy resin used by this invention. Novolak mold epoxy resins, such as bisphenol mold epoxy resins, such as the bisphenol A mold epoxy resin and a bisphenol female mold epoxy resin, a phenol novolak mold epoxy resin, and a cresol novolak mold epoxy resin, a naphthalene mold epoxy resin, a TORIFE Norian alkane mold epoxy resin, a biphenyl mold epoxy resin, a phenol aralkyl mold epoxy resin, a cyclopentadiene mold epoxy resin, etc. are illustrated especially. What has the liquid crystal structure shown with the following structure expression also in these epoxy resins is desirable.

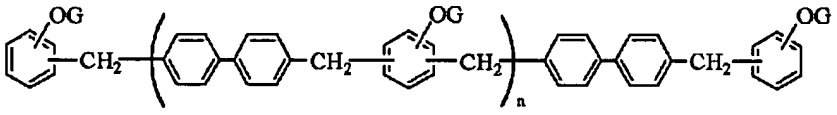
[0011]

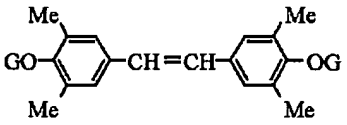
[Formula 1]











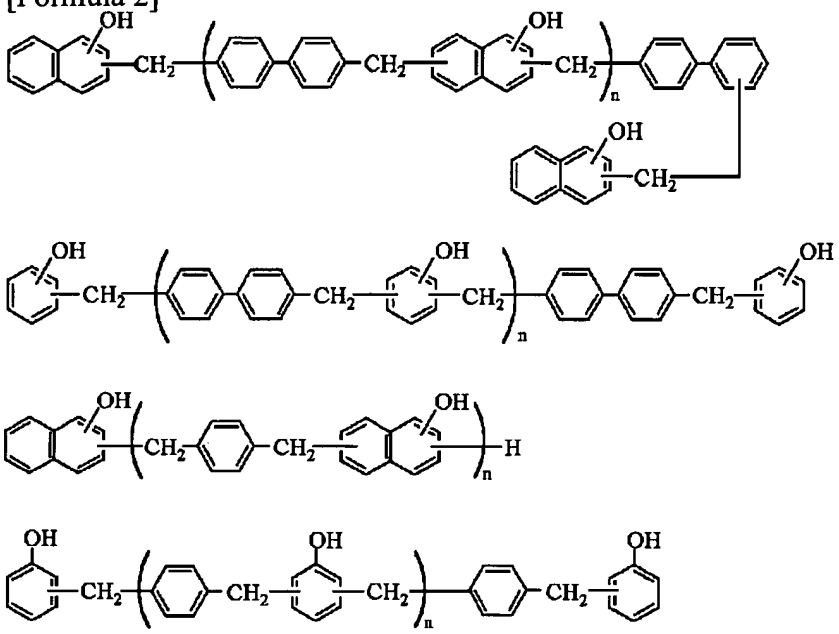
(G -- a glycidyl group and Me -- a methyl group and n -- the integer of 0-10 -- it is the integer of 1-5 preferably.)

[0012] As for especially the total chlorine content in these epoxy resins, it is desirable that it is 1000 ppm or less 1500 ppm or less. Moreover, it is desirable that extract water chlorine in 20 hours in 50% epoxy resin concentration is 5 ppm or less at 120 degrees C. If a total chlorine content exceeds 1500 ppm or extract water chlorine exceeds 5 ppm, the humidity-tolerant reliability of a semi-conductor may fall.

[0013] Anythings are usable if it is the phenolic compound (phenol resin) which contains two or more hydroxyl groups of phenol nature in 1 molecule as a curing agent of this invention. Specifically, the thing containing the phenolic hydroxyl group of bisphenol mold phenol resin, such as novolak mold phenol resin, such as phenol novolak resin and cresol novolak resin, bisphenol A, and Bisphenol F, phenol aralkyl resin, TORIFE Norian alkane mold resin, naphthalene mold phenol resin, biphenyl mold phenol resin, cyclopentadiene mold phenol resin, or the following structure etc. is illustrated.

[0014]

[Formula 2]



(n -- the integer of 0-10 -- it is the integer of 1-5 preferably.)

[0015] As for each of chlorine ions extracted at 120 degrees C, especially sodium ion, etc., it is desirable like [the above-mentioned phenol resin] an epoxy resin that it is 5 ppm or less 10 ppm or less.

[0016] The range where 0.5-1.6 mols of phenolic hydroxyl groups in phenol resin become 0.6-1.4 mols to one mol of epoxy groups in an epoxy resin is suitable for especially the operating rate of an epoxy resin and phenol resin. It may become that from which reinforcement low crosslinking density and sufficient is not obtained except that the ratio of a phenolic hydroxyl group will become high if the rate of a phenolic hydroxyl group may be insufficient for a hydroxyl group in less than 0.5 mols, the rate of homopolymerization of an epoxy group may increase, glass transition temperature may become low and it exceeds 1.6 mols, and reactivity falls.

[0017] Furthermore, it is desirable to blend a hardening accelerator in this invention. As a hardening accelerator, triphenyl phosphine, tributyl phosphine, The Tori (p-methylphenyl) phosphine, the Tori (nonylphenyl) phosphine, Organic phosphorus system compounds, such as the 4th class salts of these, such as the triphenyl phosphine triphenyl borane, and a derivative of triphenyl phosphine with which at least 2 and 6- was permuted at least for 2, 4, and 6- by the methoxy group or tetra-phenyl phosphonium tetraphenyl borate, Tertiary amine compounds, such as triethylamine, benzyl dimethylamine, and alpha-methylbenzyl dimethylamine, Diazabicycloundecen system compounds, such as 1 and 8-diazabicyclo [5.4.0] undecene -7, 6-dibutylamino -1, and 8-diazabicyclo [5.4.0] undecene-diazabicyclo [7, 1, and 4-] [2.2.2] octane (cyclo amidine system derivative), 2-methylimidazole, 4-methyl imidazole, 2-

phenylimidazole, 2-ethyl-4-methylimidazole, 2-phenyl-4-methylimidazole, Imidazole compounds, such as a 2-phenyl-4-hydroxymethyl imidazole, 2-phenyl-4-methyl-5-hydroxymethylimidazole, and 2-phenyl-4, 5-dihydroxymethylimidazole, etc. can be used. The loadings of a hardening accelerator have desirable 0.01 - 10 mass section to the total quantity 100 mass section of an epoxy resin and phenol resin.

[0018] this invention constituent is characterized by blending a spherical cristobalite as a minerals bulking agent.

[0019] A thing 75 micrometers or less is [a maximum grain size] especially here, especially desirable [mean particle diameter / 0.3-50 micrometers / 0.3-30 micrometers] especially especially as spherical fused silica as a raw material, although the above-mentioned spherical cristobalite can be obtained by the Christo labite-ization of the spherical fused silica conventionally manufactured by the well-known process at 0.3-20 micrometers 100 micrometers or less 150 micrometers or less. When mean particle diameter exceeds 50 micrometers, particle size becomes coarse too much, it becomes easy to cause gate plugging metallurgy wearing of die, and there is a case where a particle becomes fine too much and it becomes impossible for mean particle diameter to fill up with less than 0.3 micrometers so much. Moreover, if a maximum grain size exceeds 150 micrometers, particle size will become coarse too much and it will become easy to cause gate plugging metallurgy wearing of die. In addition, it can ask for a mean diameter here as a weighted mean (median size) in the particle-size-distribution measurement for example, by laser optical diffraction etc.

[0020] Moreover, as for spherical fused silica, it is desirable that the alkali metal contained as an impurity and the 20 ppm or less of the amounts of halogens are especially 10 ppm or less, respectively. If what cristobalite-ized spherical fused silica to which one of the amounts of impurities exceeds 20 ppm is used as a bulking agent, a damp-proof fall may arise. In addition, the above-mentioned alkali metal and the amount of halogens are the values which specifically measured the alkali metal and the amount of halogens of extract water after paying 10g of samples to a 250 cc plastic container, adding 100 cc of pure water, leaving it with a 95-degree C thermostat after a shaking for 30 minutes for 20 hours and separating a sample subsequently with ion chromatography.

[0021] Furthermore, in this invention, since a cristobalite crystallizes from a melting condition while the spherical fused silica of a raw material had maintained the configuration and particle size distribution, if what has the content of uranium or thorium very few especially as an impurity, and uranium and the high grade composition spherical silica whose thorium content is 1 or less ppb are used as a raw material, the suitable spherical cristobalite for memory devices can be obtained concretely.

[0022] After heating 1200-1600 degrees C of such spherical fused silica at a 1300-1500-degree C elevated temperature for 5 to 24 hours and growing up a crystal certainly, it can be made to cristobalite-ize especially by this invention by cooling to a room temperature slowly over 20 - 50 hours. In addition, the amount of 10-100-degree-C/of a programming rate is suitable from a room temperature to predetermined temperature in this case. If a long duration important point may be carried out to whenever [stoving temperature] cristobalite-izing completely at less than 1200 degrees C and it exceeds 1600 degrees C, spherical fused silica particles will weld, and there is a case where it stops maintaining the original particle size distribution. Moreover, if it may become inadequate in less than 5 hours crystallizing heating time and it exceeds 24 hours, it is sufficient time amount for crystallization, but since it must maintain at an elevated temperature for a long time, a cost rise may be caused.

[0023] Thus, since the spherical cristobalite obtained can maintain the configuration of the fused silica of a raw material, and particle size distribution as it is, it can make the fill to an epoxy resin high, and can also raise thermal conductivity. Furthermore, since the configuration is spherical, a fluidity is good and metal mold wear also has the description from which very few constituents are obtained.

[0024] A front face may be beforehand processed and used for the above-mentioned spherical cristobalite by coupling agents, such as a silane coupling agent, a titanium system coupling agent, and an aluminate coupling agent.

[0025] Minerals bulking agents other than the above-mentioned spherical cristobalite can be blended with the epoxy resin constituent of this invention as a minerals bulking agent. The spherical silica

manufactured as other minerals bulking agents with the fused silica ground, for example with the ball mill etc., the spherical silica obtained by carrying out flame fusion, a sol gel process, etc., a crystal silica, an alumina, boron nitride, CHITSU-ized aluminum, CHITSU-ized silicon, a magnesia, magnesium silicate, etc. are mentioned. In addition, in order to high-fill up an epoxy resin constituent with a minerals bulking agent, it is desirable for all the bulking agents to be used to be spherical. Especially as other minerals bulking agents, spherical fused silica is suitable.

[0026] this invention -- setting -- the above-mentioned spherical cristobalite -- 90 mass % of all minerals bulking agents -- exceeding -- the range to 100 mass %, for example, 91 to 100 mass %, -- it blends in the range of 91.4 - 100 mass % preferably. The content of the spherical cristobalite in [all] a minerals bulking agent cannot attain sufficient high temperature conduction-ization (usually 2.0 or more W/mK) to be needed for the closure of a high exoergic type semiconductor device below by 90 mass %.

[0027] The 300 - 1200 mass section especially the 500 - 1000 mass section, and division 600 - the 1000 mass sections are usually suitable for the amount of the total used of a minerals bulking agent to the total amount 100 mass section of an epoxy resin and a curing agent. When the amount of the minerals bulking agent used cannot fully raise thermal conductivity under in the 300 mass sections, water absorption also increases, a crack may go into a package at the temperature in the case of a solder reflow and the 1200 mass sections are exceeded, there is a case where viscosity becomes high too much and it becomes impossible to fabricate.

[0028] Furthermore, to this invention constituent, fumed silica, such as Aerosil, can also be added for thixotropic grant. It is better to use it, after mixing to other bulking agents and homogeneity using mixed equipments, such as a ball mill, beforehand, when using this kind of fumed silica.

[0029] Moreover, to the constituent of this invention, powder, such as well-known silicone rubber and gel, a silicone modified epoxy resin, silicone denaturation phenol resin, the thermoplastics that consists of methyl-methacrylate-styrene butadiene rubber may be added as a low stress-ized agent from the former. In addition, the addition of these resin can usually be made into an amount in the range which does not bar the effectiveness of this invention.

[0030] Furthermore, coloring agents, such as coupling agents, such as a silane coupling agent, a titanium system KASHIPU ring agent, and an aluminate coupling agent, and carbon black, the Nonion system surfactant, a fluorochemical surfactant, silicone oil, etc. get wet as an arbitration component, and an improver, a defoaming agent, etc. can be added depending on the case. In addition, the addition of these arbitration component can usually be made into an amount in the range which does not bar the effectiveness of this invention.

[0031] The epoxy resin constituent containing the spherical cristobalite of this invention can be manufactured by kneading enough with 2 rolls, continuation kneading equipment, etc., after mixing many above-mentioned raw materials to homogeneity using a high-speed mixer etc. As kneading temperature, 50-110 degrees C is desirable. An epoxy resin constituent can be manufactured by sheet-izing after kneading thinly, and cooling and grinding.

[0032] As for the hardened material of the epoxy resin constituent containing the above-mentioned spherical cristobalite, it is desirable that thermal conductivity is 2.0 or more W/mK. If thermal conductivity is smaller than 2.0 W/mK, in order to be unable to miss the heat generated from the semiconductor device by which the closure was carried out, but to delay actuation of a semiconductor device or to cause incorrect actuation, it is desirable that thermal conductivity is 2.0 or more W/mK. Moreover, when a semiconductor device contains individual semi-conductors (discrete), such as a diode transistor, since a high current flows to a semiconductor device, generation of heat of a semiconductor device is large, and it is still more desirable that thermal conductivity is 2.1 or more W/mK.

[0033]

[Effect of the Invention] When the epoxy resin constituent containing the spherical cristobalite of this invention carries out the amount combination of specification of the spherical cristobalite as a minerals bulking agent, a fluidity is good, and there is little metal mold wear and it gives the hardened material of the formation of high temperature conduction. Therefore, the epoxy resin constituent containing the spherical cristobalite of this invention can be suitably used also as an object for the latest devices as a

semi-conductor closure ingredient.
[0034]
[Example] Although the example of preparation, an example, and the example of a comparison are shown and this invention is explained concretely hereafter, this invention is not restricted to the following example. In addition, in the following example, % shows mass % and the section all shows the mass section.
[0035] After leaving the spherical fused silica which has the particle size distribution shown in the [example of preparation] table 1 at 1500 degrees C for 20 hours, at a rate of 100 degrees C/h, temperature was lowered and it cooled to 25 degrees C. After measuring the obtained spherical cristobalite with the electron microscope and checking a configuration, the crystal structure was checked according to particle size distribution and an X diffraction. Particle size distribution are shown in Table 1.
[0036]
[Table 1]

		原料球状溶融シリカ	本発明品 (球状クリストバライト)
粒度分布 (累積質量%)	96μm以下	100.0	100.0
	64μm以下	95.8	96.7
	48μm以下	88.4	90.8
	24μm以下	60.1	62.2
	12μm以下	39.9	42.9
	6μm以下	22.5	25.8
	3μm以下	13.3	14.2
	1.5μm以下	4.4	5.6
平均粒径(μm)		17.3	16.2
真比重		2.2	2.36
比表面積(m^2/g)		0.9	1.0

[0037] After carrying out mixed churning for 10 minutes with balance picking and high-speed mixing equipment with a rate which shows the component shown in the spherical cristobalite and Table 2 which were obtained by examples 1-4 and the [examples 1-4 of comparison] above in Table 2, the epoxy resin constituent was obtained by kneading with a continuation extruder.
[0038] Many following physical properties were measured per [which was obtained] epoxy resin constituent. A result is shown in Table 2.
Spiral flow: Spiral flow was measured by transfer-molding by 2 the molding temperature of 175 degrees C, and the compacting pressure of 6.9Ns/mm.
Gelation time: Time amount until an epoxy resin constituent becomes gel with a 175-degree C hot platen was measured.
Melt viscosity: Viscosity was measured at the temperature of 175 degrees C under 10kg pressurization using the nozzle with a diameter of 1mm using the quantity-ized type flow tester.
Thermal conductivity: It measured in Anter-2021 (product made from U.S. Anter).
Wire deformation: Multiplan jar equipment was used and transfer-molded by 2 using the 2 ream QFP matrix frame (measurement size = 10) the molding temperature of 175 degrees C, and the compacting pressure of 6.9Ns/mm. The fabricated QFP package was investigated using the soft X-ray apparatus, and the existence of deformation was checked.
O :-wire-deformation-less x : those with wire deformation [0039]
[Table 2]

成分（質量部）	実施例1	実施例2	実施例3	実施例4	比較例1	比較例2	比較例3	比較例4
球状クリストバライト	530	580	540	530	-	-	500	500
結晶シリカ	50	-	20	50	580	-	80	-
球状溶融シリカ	-	-	20	-	-	480	-	70
EOCN-1020-55	10	10	10	12	10	10	10	10
YX-4000H	45	45	45	47	45	45	45	45
IP-100	5	5	5	-	5	5	5	5
TD2131	33	33	33	35	33	33	33	33
三酸化アンチモン	10	10	10	10	10	10	10	10
BREN-S	7	7	7	7	7	7	7	7
カルナバワックス	1	1	1	1	1	1	1	1
トリフェニルホスフィン	1	1	1	1	1	1	1	1
スパイラルフロー(cm)	91	102	105	85	20	150	18	94
ゲル化時間(sec)	15	14	16	15	13	17	13	17
溶融粘度(Pa・s)	13	11	8	21	180	5	178	12
熱伝導率(W/mK)	2.41	2.29	2.20	2.45	2.51	0.73	2.31	1.80
ワイヤー変形	○	○	○	○	×	○	×	○

Spherical fused silica: Raw material spherical fused silica before cristobalite-izing (mean particle diameter of 17.3 micrometers)
crystal silica: -- crystallite 3K (30 micrometers of mean diameters), and made in Tatsumori -- an EOCN-1020-55:o-cresol novolak mold epoxy resin, a YXby Nippon Kayaku Co., Ltd.4000H:biphenyl mold epoxy resin, IP-100made from oil-ized Shell:indene oligomer, TDby Nippon Steel Chemical Co., Ltd.2131:novolak mold phenol resin, a Dainippon Ink BREN-S:bromination epoxy resin, and the antimony trioxide by Nippon Kayaku Co., Ltd. -- the triphenyl phosphine [by carnauba wax / by :Sumitomo Metal Mining /:Nikko Fine Products Co., Ltd.]:Hokko Chem make

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[Translation done.]

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CLAIMS

[Claim(s)]
[Claim 1] The high temperature conductivity epoxy resin constituent characterized by coming to blend a spherical cristobalite exceeding 90 mass % of the whole bulking agent as a minerals bulking agent in the epoxy resin constituent which uses an epoxy resin, a curing agent, and a minerals bulking agent as an indispensable component.
[Claim 2] The epoxy resin constituent according to claim 1 a spherical cristobalite is 0.3-50 micrometers of mean diameters, and is [constituent] the cristobalite ghost of the spherical fused silica of 150 micrometers or less of maximum grain sizes.
[Claim 3] The epoxy resin constituent according to claim 1 or 2 whose thermal conductivity of a hardened material is 2.0 or more W/mK.
[Claim 4] The epoxy resin constituent according to claim 1, 2, or 3 which is an object for the semi-conductor closures.
[Claim 5] The semiconductor device which it comes to close with the hardened material of an epoxy resin constituent according to claim 4.

[Translation done.]